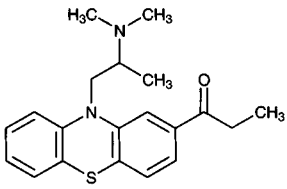
Column: 150 × 4.6 5 μm Hypersil ODS
Mobile phase: MeCN:50 mM pH 7 KH_2PO_4 buffer 30:70
Flow rate: 1
Injection volume: 100
Detector: UV 218

CHROMATOGRAM
Retention time: 4.1
Limit of detection: 20 ng/mL
Limit of quantitation: 100 ng/mL

REFERENCE
Erah,P.O.; Barrett,D.A.; Shaw,P.N. Reversed-phase high-performance liquid chromatographic assay methods for the analysis of a range of penicillins in in vitro permeation studies, *J.Chromatogr.B*, **1998**, *705*, 63–69.

Propiomazine



Molecular formula: $C_{20}H_{24}N_2OS$
Molecular weight: 340.49
CAS Registry No.: 362-29-8, 1240-15-9 (HCl)
Merck Index: 8007
Lednicer No.: 1 376

SAMPLE
Matrix: solutions
Sample preparation: Prepare a 10 μg/mL solution in MeOH, inject a 20 μL aliquot.

HPLC VARIABLES
Column: 125 × 4.9 Spherisorb S5W silica
Mobile phase: MeOH containing 10 mM ammonium perchlorate and 1 mL/L 100 mM NaOH in MeOH, pH 6.7
Flow rate: 2
Injection volume: 20
Detector: E, LeCarbone, V25 glassy carbon electrode, + 1.2 V

CHROMATOGRAM
Retention time: 2.7

OTHER SUBSTANCES
Also analyzed: acebutolol, acepromazine, acetophenazine, N-acetylprocainamide, albuterol, alprenolol, amethocaine, amiodarone, amitriptyline, antazoline, atenolol, azacyclonal, bamethan, benactyzine, benperidol, benzethidine, benzocaine, benzoctamine, benzphetamine, benzquinamide, bromhexine, bromodiphenhydramine, bromperidol, brompheniramine, brompromazine, buclizine, bufotenine, bupivacaine, buprenorphine, butacaine, butethamate, chlorcyclizine, chlorpheniramine, chlorphenoxamine, chlorprenaline, chlorpromazine, chlorprothixene, cimetidine, cinchonidine, cinnarizine, clemastine, clomipramine, clonidine, cocaine, cyclazocine, cyclizine, cyclopentamine, cyproheptadine, deserpidine, desipramine, dextromoramide, dextropropoxyphene, dicyclomine, diethylcarbamazine, diethylpropion, diethylthiambutene, dihydroergotamine, dimethindene, dimethothiazine, diphenhydramine, diphenoxylate, dipipanone, diprenorphine, dipyridamole, disopyramide, dothiepin, doxapram, doxepin, doxylamine, droperidol, ephedrine, ergocornine, ergocristine, ergocristinine, ergocryptine, ergometrine, ergosine, ergosinine, ergotamine, ethopropazine, etorphine, etoxeridine, fenethazine, fenfluramine, fenoterol, fentanyl, flavoxate, fluopromazine, flupenthixol, fluphenazine, flurazepam, haloperidol, hydroxyzine, hyoscine, ibogaine, imipramine, indapamine, iprindole, isothipendyl, isoxsuprine, ketanserin, laudanosine, lidocaine, lofepramine, loxapine, maprotiline, mecamylamine, meclophenoxate, meclozine, medazepam, mephentermine, mepivacaine, meptazinol, mepyramine, mesoridazine, metaraminol, methadone, methamphetamine, methapyrilene, methdilazene, methotrimeprazine, methoxamine, methoxyphenamine, methoxypromazine,

methylephedrine, methylergonovine, methysergide, metoclopramide, metopimazine, metoprolol, mianserin, morazone, nadolol, nalorphine, naloxone, naphazoline, nicotine, nifedipine, nomifensine, nortriptyline, noscapine, orphenadrine, oxeladin, oxprenolol, oxymetazolin, papaverine, pargyline, pecazine, penbutolol, pentazocine, penthienate, pericyazine, perphenazine, phenadoxone, phenampromide, phenazocine, phenbutrazate, phendimetrazine, phenelzine, phenglutarimide, phenindamine, pheniramine, phenmetrazine, phenomorphan, phenoperidine, phenothiazine, phenoxybenzamine, phentolamine, phenylephrine, phenyltoloxamine, physostigmine, piminodine, pimozide, pindolol, pipamazine, pipazethate, piperacetazine, piperidolate, pipradol, pirenzepine, piritramide, pizotifen, practolol, pramoxine, prazosin, prenylamine, prilocaine, primaquine, proadifen, procainamide, procaine, prochlorperazine, procyclidine, proheptazine, prolintane, promazine, promethazine, pronethalol, properidine, propranolol, prothipendyl, protriptyline, proxymetacaine, pseudoephedrine, pyrimethamine, quinidine, quinine, ranitidine, rescinnamine, sotalol, tacrine, terazosin, terbutaline, terfenadine, thenyldiamine, theophylline, thiethylperazine, thiopropazate, thioproperazine, thioridazine, thiothixene, thonzylamine, timolol, tocainide, tolpropamine, tolycaine, tranylcypromine, trazodone, trifluoperazine, trifluperidol, trimeperidine, trimeprazine, trimethobenzamide, trimethoprim, trimipramine, tripelennamine, triprolidine, tryptamine, verapamil, xylometazoline

REFERENCE

Jane,I.; McKinnon,A.; Flanagan,R.J. High-performance liquid chromatographic analysis of basic drugs on silica columns using non-aqueous ionic eluents. II. Application of UV, fluorescence and electrochemical oxidation detection, *J.Chromatogr.*, **1985**, *323*, 191–225.

SAMPLE

Matrix: solutions

HPLC VARIABLES

Column: 250 × 4.6 amylose tris(3,4,5-trimethylphenylcarbamate)
Mobile phase: Hexane:isopropanol 98:2
Flow rate: 0.5
Detector: UV

CHROMATOGRAM

Retention time: 28 (-), 41 (+)

KEY WORDS

chiral

REFERENCE

Okamoto,Y.; Aburatani,R.; Hatano,K.; Hatada,K. Optical resolution of racemic drugs by chiral HPLC on cellulose and amylose tris(phenylcarbamate) derivatives, *J.Liq.Chromatogr.*, **1988**, *11*, 2147–2163.

SAMPLE

Matrix: solutions
Sample preparation: Prepare a 1 mg/mL solution in MeOH, inject a 5 μL aliquot.

HPLC VARIABLES

Column: 250 × 4.6 5 μm Lichrosphere cyanopropyl
Mobile phase: Carbon dioxide:MeOH:isopropylamine 90:10:0.05
Column temperature: 50
Flow rate: 3
Injection volume: 5
Detector: UV 254

CHROMATOGRAM

Retention time: 2.48

OTHER SUBSTANCES

Simultaneous: triflupromazine, carphenazine, methotrimeprazine, promazine, perphenazine, thiothixene, reserpine, acetophenazine, ethopropazine, deserpidine, methotrimeprazine
Interfering: promethazine, chlorprothixene

KEY WORDS
SFC; pressure 200 bar

REFERENCE
Berger,T.A.; Wilson,W.H. Separation of drugs by packed column supercritical fluid chromatography. 1. Phenothiazine antipsychotics, *J.Pharm.Sci.*, **1994**, *83*, 281–286.

SAMPLE
Matrix: solutions

HPLC VARIABLES
Column: 250 × 4.6 Zorbax RX
Mobile phase: Gradient. A was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 1 L water. B was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 200 mL water, make up to 1 L with MeCN. A:B from 100:0 to 0:100 over 30 min, maintain at 0:100 for 5 min.
Column temperature: 30
Flow rate: 2
Detector: UV 210

OTHER SUBSTANCES
Also analyzed: acepromazine, acetaminophen, acetophenazine, albuterol, aminophylline, amitriptyline, amobarbital, amoxapine, amphetamine, amylocaine, antipyrine, aprobarbital, aspirin, atenolol, atropine, avermectin, barbital, benzocaine, benzoic acid, benzotropine, benzphetamine, berberine, bibucaine, bromazepan, brompheniramine, buprenorphine, buspirone, butabarbital, butacaine, butethal, caffeine, carbamazepine, carbromal, chloramphenicol, chlordiazepoxide, chloroquine, chlorothiazide, chloroxylenol, chlorphenesin, chlorpheniramine, chlorpromazine, chlorpropamide, chlortetracycline, cimetidine, cinchonidine, cinchonine, clenbuterol, clonazepam, clonixin, clorazepate, cocaine, codeine, colchicine, cortisone, coumarin, cyclazocine, cyclobenzaprine, cyclothiazide, cyheptamide, cymarin, danazol, danthron, dapsone, debrisoquine, desipramine, dexamethasone, dextromethorphan, dextropropoxyphene, diamorphine, diazepam, diclofenac, diethylpropion, diethylstilbestrol, diflunisal, digitoxin, digoxin, diltiazem, diphenhydramine, diphenoxylate, diprenorphine, dipyrone, disulfiram, dopamine, doxapram, doxepin, dronabinol, ephedrine, epinephrine, epinine, estradiol, estriol, estrone, ethacrynic acid, ethosuximide, etonitazene, etorphine, eugenol, famotidine, fenbendazole, fencamfamine, fenoprofen, fenproporex, fentanyl, flubendazole, flufenamic acid, flunitrazepam, 5-fluorouracil, fluoxymesterone, fluphenazine, furosemide, gentisic acid, gitoxigenin, glipizide, glunixin, glutethimide, glybenclamide, guaiacol, halazepam, haloperidol, hydrochlorothiazide, hydrocodone, hydrocortisone, hydromorphone, hydroxyquinoline, ibogaine, ibuprofen, iminostilbene, imipramine, indomethacin, isocarbostyril, isocarboxazid, isoniazid, isoproterenol, isoxsuprine, ivermectin, ketamine, ketoprofen, kynurenic acid, levorphanol, lidocaine, lorazepam, lormetazepam, loxapine, mazindol, mebendazole, meclizine, meclofenamic acid, medazepam, mefenamic acid, megestrol, mepacrine, meperidine, mephentermine, mephenytoin, mephesin, mephobarbital, mepivacaine, mescaline, mesoridazine, methadone, methamphetamine, methapyrilene, methaqualone, methazolamide, methocarbamol, methoxamine, methsuximide, methyl salicylate, methyldopa, methyldopamine, methylphenidate, methylprednisolone, methyltestosterone, methyprylon, metoprolol, mibolerone, morphine, nadolol, nalorphine, naloxone, naltrexone, naphazoline, naproxen, nefopam, niacinamide, nicotine, niacin, nifedipine, niflumic acid, nitrazepam, norepinephrine, nortriptyline, noscapine, nylidrin, oxazepam, oxycodone, oxymorphone, oxyphenbutazone, oxytetracycline, papaverine, pargyline, pemoline, pentazocine, pentobarbital, persantine, phenacetin, phenazocine, phenazopyridine, phencyclidine, phendimetrazine, phenelzine, pheniramine, phenobarbital, phenothiazine, phensuximide, phentermine, phenylbutazone, phenylephrine, phenylpropanolamine, piperocaine, prazepam, prednisolone, primidone, probenecid, propranolol, propylparaben, pseudoephedrine, puromycin, pyrilamine, pyrithyldione, quazepam, quinaldic acid, quinidine, quinine, ranitidine, recinnamine, reserpine, resorcinol, saccharin, albuterol, salicylamide, salicylic acid, scopolamine, scopoletin, secobarbital, strychnine, sulfacetamide, sufadiazine, sulfadimethoxine, sulfaethidole, sulfamerazine, sulfamethazine, sulfamethoxizole, sulfanilamide, sulfapyridine, sulfasoxizole, sulindac, tamoxifen, temazepam, testosterone, tetracaine, tetracycline, tetramisole, thebaine, theobromine, theophylline, thiabendazole, thiamine, thiamylal, thiobarbituric acid, thioridazine, thiosalicylic acid, thiothixene, thymol, tolazamide, tolazoline, tobutamide, tolmetin, tranylcypromine, triamcinolone, tribenzylamine, trichloromethiazide, trifluoperazine, trihexyphenidyl, trimethoprim, tripelennamine, triprolidine, tropacocaine, tyramine, verapamil, vincamine, warfarin, yohimbine, zoxazolamine

REFERENCE
Hill,D.W.; Kind,A.J. Reversed-phase solvent gradient HPLC retention indexes of drugs, *J.Anal.Toxicol.*, **1994**, *18*, 233–242.

SAMPLE
Matrix: solutions

HPLC VARIABLES
Column: 250 × 4.6 5 μm Supelcosil LC-DP (A) or 250 × 4 5 μm LiChrospher 100 RP-8 (B)
Mobile phase: MeCN:0.025% phosphoric acid:buffer 25:10:5 (A) or 60:25:15 (B) (Buffer was 9 mL concentrated phosphoric acid and 10 mL triethylamine in 900 mL water, adjust pH to 3.4 with dilute phosphoric acid, make up to 1 L.)
Flow rate: 0.6
Injection volume: 25
Detector: UV 229

CHROMATOGRAM
Retention time: 14.08 (A), 7.10 (B)

OTHER SUBSTANCES
Also analyzed: acebutolol, acepromazine, acetaminophen, acetazolamide, acetophenazine, albuterol, alprazolam, amitriptyline, amobarbital, amoxapine, antipyrine, atenolol, atropine, azatadine, baclofen, benzocaine, bromocriptine, brompheniramine, brotizolam, bupivacaine, buspirone, butabarbital, butalbital, caffeine, carbamazepine, cetirizine, chlorcyclizine, chlordiazepoxide, chlormezanone, chloroquine, chlorpheniramine, chlorpromazine, chlorpropamide, chlorprothixene, chlorthalidone, chlorzoxazone, cimetidine, cisapride, clomipramine, clonazepam, clonidine, clozapine, cocaine, codeine, colchicine, cyclizine, cyclobenzaprine, dantrolene, desipramine, diazepam, diclofenac, diflunisal, diltiazem, diphenhydramine, diphenidol, diphenoxylate, dipyridamole, disopyramide, dobutamine, doxapram, doxepin, droperidol, encainide, ethidium bromide, ethopropazine, fenoprofen, fentanyl, flavoxate, fluoxetine, fluphenazine, flurazepam, flurbiprofen, fluvoxamine, furosemide, glutethimide, glyburide, guaifenesin, haloperidol, homatropine, hydralazine, hydrochlorothiazide, hydrocodone, hydromorphone, hydroxychloroquine, hydroxyzine, ibuprofen, imipramine, indomethacin, ketoconazole, ketoprofen, ketorolac, labetalol, levorphanol, lidocaine, loratadine, lorazepam, lovastatin, loxapine, mazindol, mefenamic acid, meperidine, mephenytoin, mepivacaine, mesoridazine, metaproterenol, metformin, methadone, methdilazine, methocarbamol, methotrexate, methotrimeprazine, methoxamine, methyldopa, methylphenidate, metoclopramide, metolazone, metoprolol, metronidazole, midazolam, moclobemide, morphine, nadolol, nalbuphine, naloxone, naphazoline, naproxen, nifedipine, nizatidine, norepinephrine, nortriptyline, oxazepam, oxycodone, oxymetazoline, paroxetine, pemoline, pentazocine, pentobarbital, pentoxifylline, perphenazine, pheniramine, phenobarbital, phenol, phenolphthalein, phentolamine, phenylbutazone, phenyltoloxamine, phenytoin, pimozide, pindolol, piroxicam, pramoxine, prazepam, prazosin, probenecid, procainamide, procaine, prochlorperazine, procyclidine, promazine, promethazine, propafenone, propantheline, propofol, propranolol, protriptyline, quazepam, quinidine, quinine, racemethorphan, ranitidine, remoxipride, risperidone, salicylic acid, scopolamine, secobarbital, sertraline, sotalol, spironolactone, sulfinpyrazone, sulindac, temazepam, terbutaline, terfenadine, tetracaine, theophylline, thiethylperazine, thiopental, thioridazine, thiothixene, timolol, tocainide, tolbutamide, tolmetin, trazodone, triamterene, triazolam, trifluoperazine, triflupromazine, trimeprazine, trimethoprim, trimipramine, verapamil, warfarin, xylometazoline, yohimbine, zopiclone

KEY WORDS
details of plasma extraction

REFERENCE
Koves,E.M. Use of high-performance liquid chromatography-diode array detection in forensic toxicology, *J.Chromatogr.A*, **1995**, *692*, 103–119.

SAMPLE
Matrix: tissue
Sample preparation: Condition a Sep-Pak C18 SPE cartridge with 5 mL MeOH and 5 mL water. Homogenize kidney with a kitchen grinder. Weigh out a 5 g sample and add 20 mL MeCN with

continuous gentle mixing, mix vigorously on a vibromixer at 1500 rpm for 30 s, sonicate for 2 min, centrifuge at 4000 g for 5 min. Mix 7.5 mL sample extract and 40 mL 10% NaCl and add to SPE cartridge, wash with 1 mL 10 mM sulfuric acid, wash with 2 mL air, elute with 2 mL acidic MeCN. Place eluate in a washed tube and evaporate to 300 μL at 70° under a stream of nitrogen, mix gently, add 1 mL n-hexane, mix on a vibromixer for 30 s, centrifuge at 2000 g, inject a 50 μL aliquot of the aqueous phase. (Acidic MeCN was 1 mL 50 mM sulfuric acid and 100 mL MeCN. The washed tube was prepared by rinsing with concentrated ammonia, water, and acetone and drying under a stream of nitrogen.)

HPLC VARIABLES
Guard column: 10 × 2.1 37-50 μm Bondapak C18
Column: 300 × 3.9 Bondapak C18
Mobile phase: MeCN:water 55:45 containing 2.46 g/L anhydrous sodium acetate, pH adjusted to 6.5 with acetic acid
Flow rate: 1.2
Injection volume: 50
Detector: UV 240

CHROMATOGRAM
Retention time: 18
Limit of detection: 4 ng/g

OTHER SUBSTANCES
Extracted: azaperol, carazolol, acepromazine, xylazine, azaperone, haloperidol, chlorpromazine

KEY WORDS
SPE; pig; kidney

REFERENCE
Keukens,H.J.; Aerts,M.M.L. Determination of residues of carazolol and a number of tranquilizers in swine kidney by high-performance liquid chromatography with ultraviolet and fluorescence detection, *J.Chromatogr.*, **1989**, *464*, 149–161.

SAMPLE
Matrix: tissue
Sample preparation: Condition a Bond-Elut C18 SPE cartridge with 5 mL MeOH and 5 mL water. Cut pig kidney or liver into small pieces and homogenize. 5 g Homogenate + 10 mL MeCN, shake, vortex for 30 s, sonicate for 3 min, vortex for 30 s, sonicate for 3 min, centrifuge at 10000 g for 20 min. Add 7.5 mL supernatant + 40 mL 10% NaCl to the SPE cartridge at about 1 mL/min, do not allow cartridge to dry out, wash with 850 μL 10 mM sulfuric acid, dry with air, elute with 3.5 mL acidic MeCN. Evaporate the eluate to dryness under a stream of nitrogen at 50°, reconstitute the residue in 300 μL 10 mM sulfuric acid, vortex briefly, add 1 mL hexane, vortex for 30 s, centrifuge at 2000 g for 5 min, inject an aliquot of the aqueous layer. (Acidic MeCN was 1 mL 50 mM sulfuric acid in 100 mL MeCN.)

HPLC VARIABLES
Guard column: Hypersil 5 μm SAS C1
Column: 250 mm long 5 μm Hypersil SAS C1
Mobile phase: MeCN:water 50:50 containing 0.77 g/L ammonium acetate
Flow rate: 2
Detector: E, ESA Model 5100A Coulochem, first electrode +0.4 V, second electrode (which was monitored) +0.7 V, Model 5020 guard cell after pump but before injector at +0.75 V

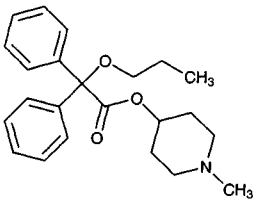
CHROMATOGRAM
Retention time: 25
Limit of detection: 2 ng/g

OTHER SUBSTANCES
Extracted: azaperol, acepromazine, carazolol, azaperone, xylazine, haloperidol, chlorpromazine

KEY WORDS
SPE; pig; kidney; liver

REFERENCE
Rose,M.D.; Shearer,G. Determination of tranquilisers and carazolol residues in animal tissue using high-performance liquid chromatography with electrochemical detection, *J.Chromatogr.*, **1992**, *624*, 471–477.

Propiverine



Molecular formula: $C_{23}H_{29}NO_3$
Molecular weight: 367.49
CAS Registry No.: 60569-19-9
Merck Index: 8018

SAMPLE
Matrix: blood, perfusate, tissue
Sample preparation: Homogenize skin with EtOH:water 70:30. 100 μL Plasma, perfusate, or skin homogenate + 200 μL 2.5 μg/mL phenytoin in MeCN, mix, centrifuge. Filter (0.45 μm) the supernatant, inject a 10 μL aliquot of the filtrate.

HPLC VARIABLES
Column: 150 × 4.6 5 μm Inertsil ODS
Mobile phase: MeCN:10 mM pH 3.5 phosphate buffer 38:62 containing 0.6% nonylamine
Injection volume: 10
Detector: UV 200

CHROMATOGRAM
Internal standard: phenytoin

OTHER SUBSTANCES
Extracted: terodiline

KEY WORDS
rat; plasma; skin

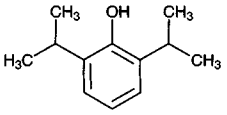
REFERENCE
Ogiso,T.; Iwaki,M.; Hirota,T.; Tanino,T.; Muraoka,O. Comparison of the in vitro skin penetration of propiverine with that of terodiline, *Biol.Pharm.Bull.*, **1995**, *18*, 968–875.

SAMPLE
Matrix: solutions

HPLC VARIABLES
Guard column: 50 × 4.6 10 μm LiChrosorb RP-18
Column: 200 × 4.6 10 μm LiChrosorb RP-18
Mobile phase: MeOH:water 60:40
Flow rate: 1.5
Detector: UV 254

REFERENCE
Göber,B.; Dressler,K.; Franke,P.; Alder,L. Zur Analytik und Stabilität von Propiverinhydrochlorid (Mictonorm) [Analysis and stability of propiverine hydrochloride (Mictonorm)], *Pharmazie*, **1986**, *41*, 840–842.

Propofol



Molecular formula: $C_{12}H_{18}O$
Molecular weight: 178.27
CAS Registry No.: 2078-54-8
Merck Index: 8020

SAMPLE

Matrix: blood

Sample preparation: To 2 mL whole blood or plasma containing 20 μL thymol in MeOH, solid blood elements obtained by centrifuging 2 mL blood containing 20 μL thymol in MeOH, solid blood elements without plasma (obtained by centrifuging 2 mL blood containing 20 μL thymol in MeOH and by washing with four portions of 0.9% NaCl), or lysed solid elements (obtained by centrifuging 2 mL blood containing 20 μL thymol in MeOH and by washing with four portions of 0.9% NaCl, and mixing with three volumes of water), add 1 mL 100 mM NaH_2PO_4 and 5 mL cyclohexane. Shake vigorously at 200 rpm for 15 min, centrifuge at 1200 g for 5 min. Add 50 μL tetraethylammonium hydroxide solution to ca. 5 mL of the cyclohexane layer, evaporate to dryness under a stream of nitrogen, reconstitute the residue in mobile phase, inject an aliquot. (Prepare tetraethylammonium hydroxide solution by mixing a 25% solution of tetraethylammonium hydroxide in EtOH with EtOH in the ratio of 3:37.)

HPLC VARIABLES

Column: 250 × 4 RP octadecyl silane (prepared as described in J. Chromatogr. Sci. 1995, 33, 377-382)
Mobile phase: MeCN:buffer 67:33 (Buffer was water adjusted to pH 4.0 with acetic acid.)
Detector: UV (wavelength not given)

CHROMATOGRAM

Retention time: 12
Internal standard: thymol (7.5)

KEY WORDS

whole blood; plasma

REFERENCE

Dawidowicz,A.J.; Fijalkowska,A. Possibilities of propofol analysis in various blood components by means of HPLC, *J.Liq.Chromatogr.Rel.Technol.*, **1996**, *19*, 1423–1435.

SAMPLE

Matrix: blood

Sample preparation: Add 30 μL 10 ng/mL or 70 μL 100 ng/mL methyldopa solution and 50 μL 2 M HCl to 50 μL plasma, make up to 170 μL with water, centrifuge at 1400 rpm for 5 min, inject an aliquot of the supernatant.

HPLC VARIABLES

Column: 100 × 8 4 μm NovaPak C18
Mobile phase: MeCN:MeOH:10 mM pH 3 sodium acetate buffer 47.25:15.75:37
Flow rate: 2
Detector: F ex 276 em 310

CHROMATOGRAM

Retention time: 8.20
Internal standard: methyldopa (5.15)

OTHER SUBSTANCES

Noninterfering: albuterol, ampicillin, amoxicillin, amphotericin B, bleomycin, ceftazidime, cefoxitin, cephalexin, ciprofloxacin, dobutamine, dopamine, epinephrine, erythromycin, esmolol, fluconazole, gentamicin, labetalol, metoclopramide, miconazole, nitroglycerin, nitroprusside, norepinephrine, paclitaxel, penicillin G benzathine, ranitidine, streptomycin, tetracycline

KEY WORDS
plasma

REFERENCE
el-Yazigi,A.; Hussein,R.F. Microdetermination of propofol in plasma by a rapid and sensitive liquid chromatographic method, *J.Pharm.Biomed.Anal.*, **1996**, *15*, 99–104.

SAMPLE
Matrix: blood, tissue
Sample preparation: Plasma, serum. 20-100 μL Plasma or serum + 100-200 μL IS in MeOH:water 50:50. Make up to 1 mL with water. Add 4 mL pentane, rock at 40 cycles per min for 15 min, centrifuge at 1500 g for 5-10 min. Remove the organic layer and add it to 1 mL 100 mM HCl, rock for 10 min, centrifuge at 1500 g for 5 min. Remove the organic layer and add it to 500 μL isopropanol:100 mM NaOH 90:10, evaporate to dryness under a stream of nitrogen, reconstitute the residue in 140-240 μL mobile phase. Add 10 μL 600 mM phosphoric acid, inject an aliquot. Tissue. Homogenize 0.5-1.5 g tissue divided into 2 × 2 mm pieces with MeCN:50 mM pH 7.0 phosphate buffer 50:50 equal to four times the tissue weight. Mix the homogenate from 20-100 μg tissue with 100 μL 500 mM pH 7.0 phosphate buffer and 100-200 μL IS in MeOH:water 50:50. Make up to a total volume 1 mL with water. Add 4 mL pentane, rock at 40 cycles per min for 15 min, centrifuge at 1500 g for 5-10 min. Remove the organic layer and add it to 1 mL 100 mM HCl, rock for 10 min, centrifuge at 1500 g for 5 min. Remove organic layer and add it to 500 μL isopropanol:100 mM NaOH 90:10, evaporate to dryness under a stream of nitrogen, reconstitute the residue in 490 μL mobile phase. Add 10 μL 600 mM phosphoric acid, inject an aliquot. Fat, skin. 100 mg fat or skin + 20 mL 5 mM sodium deoxycholate + 1 mL 500 mM phosphate buffer + 150 μL 10 (skin) or 100 (fat) μg/mL IS in MeOH:water 50:50, homogenize, steam-distill immediately at a rate of 2 mL/min. Collect 8 mL distillate in a tube containing 8 mL pentane, add 4 mL pentane, rock on a LabQuake for 15 min, centrifuge at 1500 g for 10 min. Remove the organic layer and add it to 2 mL 500 mM NaOH, rock for 10 min, centrifuge at 1500 g for 5 min. Remove the organic layer and evaporate it to about 4-5 mL under a stream of nitrogen, add 500 μL isopropanol:100 mM NaOH 90:10, evaporate to dryness under a stream of nitrogen, reconstitute the residue in 990 μL mobile phase and 10 μL 600 mM phosphoric acid for skin and 10 mL mobile phase and 10 μL 600 mM phosphoric acid for fat, inject an aliquot.

HPLC VARIABLES
Column: 100 × 4.6 3 μm Rainin Microsorb MV phenyl RP
Mobile phase: MeOH:50 mM pH 2.8 phosphate buffer 60:40
Column temperature: 40
Flow rate: 0.8
Injection volume: 50-100
Detector: E, ESA Coulochem model 5100A, Model 5020 guard cell +850 mV, Model 5010 dual analytical cell, detector 1 +300 m V, detector 2 +800 m V (monitored)

CHROMATOGRAM
Retention time: 9-10
Internal standard: 2,6-tert-butylmethylphenol (7.5-8)
Limit of quantitation: 5 ng/mL (serum), 50 ng/g (tissue)

KEY WORDS
human; plasma; serum; rat ; fat; skin; liver; stomach; intestine; pharmacokinetics

REFERENCE
Dowrie; R.H.; Ebling; W.F.; Mandema; J.W.; Stanski; D.R. High-performance liquid chromatographic assay of propofol in human and rat plasma and fourteen rat tissues using electrochemical detection, *J.Chromatogr.B.*, **1996**, *678*, 279–288.

SAMPLE
Matrix: formulations
Sample preparation: Dilute equal volume 10 mg/mL propofol and 25 mg/mL thiopental injections 1:200 with water, inject a 20 μL aliquot.

HPLC VARIABLES
Column: 150 × 4.6 5 μm Zorbax SB
Mobile phase: MeCN:buffer 45:55 (Buffer was 10 mM KH_2PO_4, adjusted to pH 4.0 with 10% phosphoric acid.)
Flow rate: 1
Injection volume: 20
Detector: UV 235

CHROMATOGRAM
Retention time: 12

OTHER SUBSTANCES
Simultaneous: thiopental

KEY WORDS
stability-indicating; injections

REFERENCE
Chernin,E.L.; Stewart,J.T.; Smiler,B. Stability of thiopental sodium and propofol in polypropylene syringes at 23 and 4°C, *Am.J.Health-Syst.Pharm.*, **1996**, *53*, 1576–1579.

SAMPLE
Matrix: solutions
Sample preparation: Inject a 20 μL aliquot of a solution in 0.9% sodium chloride.

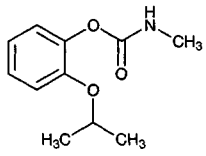
HPLC VARIABLES
Column: 150 × 4.6 5 μm Zorbax SB phenyl (A) or 300 × 4.6 10 μm T-Bondapak phenyl (B)
Mobile phase: MeCN:buffer 45:55 (A) or 50:50 (B) (Buffer was 10 mM KH_2PO_4 adjusted to pH 4.0 with 10% phosphoric acid)
Flow rate: 1
Injection volume: 20
Detector: UV 235 (A), UV 268 (B)

CHROMATOGRAM
Retention time: 12.0 (A), 10 (B)
Limit of detection: 1210 ng/mL (A), 117 ng/mL (B)

OTHER SUBSTANCES
Simultaneous: ondansetron (B), thiopental (A)

REFERENCE
King,D.T.; Stewart,J.T.; Venkateshwaran,T.G. HPLC determination of propofol-thiopental sodium and propofol-ondansetron mixtures, *J.Liq.Chromatogr.Rel.Technol.*, **1996**, *19*, 2285–2294.

Propoxur



Molecular formula: $C_{11}H_{15}NO_3$
Molecular weight: 209.25
CAS Registry No.: 114-26-1
Merck Index: 8022

SAMPLE

Matrix: blood, tissue
Sample preparation: Homogenize tissue with an equal volume of water, treat with a saturated solution of calcium chloride, let stand overnight, filter. Extract filtrate, blood, or other body fluid with an equal volume of ether. Adjust pH of aqueous layer to 2 with 2 M HCl, extract with an equal volume of ether. Combine the ether layers, evaporate to dryness, reconstitute in a suitable solvent, inject an aliquot.

HPLC VARIABLES

Column: 250 × 4.6 5 μm Zorbax cyano
Mobile phase: Iso-octane:ethyl acetate 80:20
Flow rate: 1
Injection volume: 20
Detector: RI

CHROMATOGRAM

Retention time: 7.56
Limit of detection: 100 ng

OTHER SUBSTANCES

Extracted: methyl parathion, dichlorvos, monocrotophos, quinalphos, malathion, phosphamidon, carbaryl

KEY WORDS

liver; lung

REFERENCE

Sharma,V.K.; Jadhav,R.K.; Rao,G.J.; Saraf,A.K.; Chandra,H. High performance liquid chromatographic method for the analysis of organophosphorus and carbamate pesticides, *Forensic Sci.Int.*, **1990**, *48*, 21–25.

SAMPLE

Matrix: blood, urine
Sample preparation: Add 1 mL whole blood or urine to Toxi-Tube A (Toxi-Lab, Irvine CA), add 3 mL water, mix by gentle inversion for 5 min, centrifuge at 1500 g for 5 min. Remove the organic layer and evaporate it to dryness under a stream of nitrogen at 40°, reconstitute the residue with 50 μL MeCN:water 50:50, vortex for 10 s, centrifuge at 7500 g for 2 min, inject a 10 (urine) or 30 (blood) μL aliquot. (The detector wavelength shown is the wavelength of maximum absorbance. This will not necessarily be the optimal wavelength for the separation. Multiple wavelengths from 200-350 nm can be scanned using a diode-array detector. Otherwise, 220 nm may be a reasonable choice for initial work. Matrix may interfere.)

HPLC VARIABLES

Guard column: 20 mm long Symmetry C18
Column: 250 × 4.6 5 μm Symmetry C8 (Waters)
Mobile phase: Gradient. A was 50 mM pH 3.8 sodium phosphate buffer. B was MeCN. A:B 85:15 for 6.5 min, 65:35 for 18.5 min, 20:80 for 3 min (step gradient), re-equilibrate at initial conditions for 7 min.
Column temperature: 30
Flow rate: 1 for 6.5 min, to 1.5 over 18.5 min, maintain at 1.5 for 3 min (re-equilibrate at 1.5 mL/min)
Injection volume: 10-30
Detector: UV 200.5

CHROMATOGRAM
Retention time: 17.293

KEY WORDS
whole blood

REFERENCE
Gaillard,Y.; Pépin,G. Use of high-performance liquid chromatography with photodiode-array UV detection for the creation of a 600-compound library. Application to forensic toxicology, *J.Chromatogr.A*, **1997**, *763*, 149–163.

SAMPLE
Matrix: fruit, vegetables
Sample preparation: Homogenize (Omni-Mixer) 100 g chopped sample with 250 mL MeOH at half-speed for 5 min, filter (Whatman No. 1 PS paper), make up filtrate to 500 mL with MeOH. Remove 100 mL filtrate and add it to 125 mL 4% aqueous sodium sulfate, shake well, extract mixture with 75, 50, and 50 mL portions of dichloromethane with 30 s shaking each time, drain organic layers through anhydrous sodium sulfate. Combine the organic layers and evaporate them to 1 mL under reduced pressure at 30°, transfer residue to a tube with two 2 mL rinses of dichloromethane:cyclohexane 50:50, make volume up to 10 mL with dichloromethane:cyclohexane 50:50, filter (0.45 μm), add 5 mL to a 600 × 25 tube containing 60 g 200-400 mesh BioBeads SX-3 resin (Analytical BioChemistry Laboratories), pump through at 5 mL/min with dichloromethane:cyclohexane 50:50 mobile phase, discard mobile phase for 24 min, collect fraction containing the compound for 12 min, evaporate under reduced pressure at 30° to low volume, add 15 mL MeOH, evaporate to about 1 mL, filter (0.45 μm), inject a 20 μL aliquot. Alternatively, run output from BioBeads column through a column containing 0.5 g of a mixture of Nuchar S-N(Fisher):Celite 545 1:4, at the end of the chromatography elute this column with 10 mL MeCN:toluene 75:25, evaporate the eluate under reduced pressure at 30° to low volume, add 15 mL MeOH, evaporate to about 1 mL, filter (0.45 μm), inject a 20 μL aliquot.

HPLC VARIABLES
Guard column: 50 × 4.6 Pellicular ODS (Whatman)
Column: 250 × 4.6 5 μm Apex ODS (Jones Chromatography)
Mobile phase: Gradient. MeOH:water from 10:90 to 90:10 over 23 min, to 10:90 over 4 min, re-equilibrate at 10:90 for 10 min
Flow rate: 1
Injection volume: 20
Detector: F ex 340 em 455, following post-column derivatization. The column effluent is mixed with 200 mM NaOH at 0.8 mL/min and the mixture flows through a 1 mL coil at 95° and is mixed with 500 mg/L o-phthalaldehyde and 1 mL/L 2-mercaptoethanol in 50 mM sodium tetraborate pumped at 0.8 mL/min. The mixture flows through a 0.5 mL coil at ambient temperature to the detector.

CHROMATOGRAM
Retention time: 24
Limit of detection: 5-10 ppb

OTHER SUBSTANCES
Extracted: oxamyl, methomyl, aldicarb, carbaryl, carbofuran, methiocarb

KEY WORDS
apples; broccoli; cabbage; cauliflower; potatoes; post-column reaction

REFERENCE
Chaput,D. Simplified multiresidue method for liquid chromatographic determination of N-methyl carbamate insecticides in fruits and vegetables, *J.Assoc.Off.Anal.Chem.*, **1988**, *71*, 542–546.

SAMPLE
Matrix: fruit, vegetables
Sample preparation: Blend (Waring) 100 g chopped fruit or vegetable with 200 mL acetone at low speed for 1 min, filter. 80 mL Filtrate + 100 mL petroleum ether + 100 mL dichloromethane,

shake vigorously, filter the organic phase through anhydrous sodium sulfate. Saturate the aqueous phase with 7 g NaCl, extract with 100 mL dichloromethane, filter the organic layer through anhydrous sodium sulfate. Wash the anhydrous sodium sulfate with 50 mL dichloromethane, combine the organic layers, evaporate to about 4 mL through a Snyder column on a steam bath, add 40 μL 1 mg/mL IS solution, adjust volume to 4 mL, inject a 50 μL aliquot.

HPLC VARIABLES

Column: 220 × 4.6 5 μm Spheri-5 C18
Mobile phase: Gradient. MeCN:water:13 mM ammonium acetate from 20:65:15 to 80:5:15 over 30 min.
Flow rate: 1
Injection volume: 50
Detector: MS, Vestec Model 301 thermospray, positive ion discharge mode, vaporizer tip 225-235°, SIM, m/z 210

CHROMATOGRAM

Retention time: 17
Internal standard: 2-fluoro-9-fluorenone (25)
Limit of detection: 0.25 ppm

OTHER SUBSTANCES

Extracted: aldicarb, aldicarb sulfoxide, bufencarb, carboxin, chlorbromuron, diuron, linuron, methiocarb, methomyl, metobromuron, monuron, neburon, oxamyl, thiodicarb

KEY WORDS

apples; beans; lettuce; peppers; potatoes; tomatoes

REFERENCE

Liu,C.-H.; Mattern,G.C.; Yu,X.; Rosen,R.T.; Rosen,J.D. Multiresidue determination of nonvolatile and thermally labile pesticides in fruits and vegetables by thermospray liquid chromatography/mass spectrometry, *J.Agric.Food Chem.*, **1991**, *39*, 718–723.

SAMPLE

Matrix: solutions
Sample preparation: Pass 100 mL water through column A at 5 mL/min then elute the contents of column A onto column B with the mobile phase, elute column B with the mobile phase and monitor the effluent from column B.

HPLC VARIABLES

Column: A 30 × 4.6 5 μm Spherisorb ODS C18; B 250 × 4.6 5 μm Supelcosil LC-8 C8
Mobile phase: Gradient. MeCN:water 30:70 for 5 min, to 60:40 over 10 min, maintain at 60:40 for 10 min, to 30:70 over 5 min, maintain at 30:70 for 5 min and inject next sample.
Flow rate: 1.5
Injection volume: 100000
Detector: UV 220

CHROMATOGRAM

Retention time: 16.00
Limit of detection: 65 pg/mL

OTHER SUBSTANCES

Simultaneous: carbaryl, carbofuran, captan, propham, chloropropham, barban, butylate

KEY WORDS

water; drinking water; column-switching

REFERENCE

Marvin,C.H.; Brindle,I.D.; Hall,C.D.; Chiba,M. Development of an automated high-performance liquid chromatographic method for the on-line pre-concentration and determination of trace concentrations of pesticides in drinking water, *J.Chromatogr.*, **1990**, *503*, 167–176.

SAMPLE
Matrix: solutions
Sample preparation: Condition a 10 × 4 55 mg 40 μm C18/OH Bondesil SPE cartridge (Varian/Analytichem) with 1 mL MeOH and 1 mL water, pass through 5 mL test water at 1 mL/min, pass through 500 μL pure water, elute the contents of the SPE cartridge onto the analytical column with mobile phase.

HPLC VARIABLES
Guard column: 10 × 4 4 μm Supersphere RP-8 (Merck)
Column: 250 × 4 4 μm Supersphere RP-8 (Merck)
Mobile phase: Gradient. A was MeCN:water 20:80 containing 2.5 mM sodium acetate. B was MeOH:water 20:80 containing 2.5 mM sodium acetate. C was MeCN:water 60:40 containing 2.5 mM sodium acetate. A:B:C 75:25:0 for 5 min, to 0:0:100 over 20 min, maintain at 0:0:100 for 5 min, re-equilibrate at initial conditions for 15 min.
Column temperature: 35
Flow rate: 0.75
Injection volume: 100
Detector: F ex 340 em 445 following post-column reaction. The column effluent flowed through a 50 × 4 Aminex A-27 (Bio-Rad) column at 120-140° and was mixed with reagent pumped at 1 mL/min, this mixture flowed through a 200 × 0.12 PTFE tube to the detector. (Reagent was prepared by adding 2 mL 25 mg/mL o-phthalaldehyde in MeCN and 100 μL 2-mercaptoethanol to 200 mL 5 mg/mL disodium tetraborate in water then making up to 250 mL with water.)

CHROMATOGRAM
Retention time: 23.09
Internal standard: trimethacarb (26.12)
Limit of detection: 0.03-0.05 ng/mL

OTHER SUBSTANCES
Simultaneous: aldicarb, bendiocarb, bufencarb, butocarboxim, carbanolate, carbaryl, carbofuran, cloethocarb, dioxacarb, ethiofencarb, fenobucarb, isoprocarb, methiocarb, methomyl, oxamyl, promecarb, thiofanox, tranid

KEY WORDS
water; SPE; post-column reaction

REFERENCE
Hiemstra,M.; de Kok,A. Determination of N-methylcarbamate pesticides in environmental water samples using automated on-line trace enrichment with exchangeable cartridges and high-performance liquid chromatography, *J.Chromatogr.A*, **1994**, *667*, 155–166.

SAMPLE
Matrix: solutions
Sample preparation: Flush column A with 5 mL MeOH and 5 mL MeOH:pH 5.0 ammonium acetate, pass a 100 mL sample through the column at 4 mL/min, backflush the contents of column A onto column B and start the gradient, monitor the effluent from column B.

HPLC VARIABLES
Column: A 10 × 2 15-25 μm PLRP-S styrene-divinylbenzene co-polymer (Spark Holland); B 200 × 4 5 μm Spherisorb ODS2
Mobile phase: MeOH:100 mM pH 5.0 ammonium acetate from 30:70 to 88:12 over 34 min.
Column temperature: 40
Flow rate: 0.4
Injection volume: 100000
Detector: UV 280 or MS, Hewlett-Packard 5989 A, dual EI/chemical ionization source, ion source block 250°, quadrupole 100°, m/z 64-400, desolvation chamber 65°, helium nebulizer 50 psi, second-stage momentum separator 0.5 Torr, ion source chamber 15 μTorr

CHROMATOGRAM
Retention time: 20
Limit of detection: <1 ng/mL

OTHER SUBSTANCES
Simultaneous: carbaryl, aldicarb, atrazine, barban, carbofuran, cyanazine, diuron, fluometuron, linuron, methomyl, monuron, oxamyl, simazine

KEY WORDS
water; column-switching

REFERENCE
Marcé,R.M.; Prosen,H.; Crespo,C.; Calull,M.; Borrull,F.; Brinkman,U.A.T. On-line trace enrichment of polar pesticides in environmental waters by reversed-phase liquid chromatography-diode array detection-particle beam mass spectrometry, *J.Chromatogr.A*, **1995**, *696*, 63–74.

SAMPLE
Matrix: solutions
Sample preparation: Inject a 20 μL aliquot.

HPLC VARIABLES
Guard column: 10 × 3 5 μm Kromasil-100-C18 (Akzo Nobel)
Column: 150 × 4 5 μm Kromasil-100-C18 (Akzo Nobel)
Mobile phase: MeCN:buffer 28:72 (Buffer was 820 mg/L sodium tetraborate decahydrate containing 50 μg/mL phthalaldehyde and 0.06 μL/mL 2-mercaptoethanol, adjusted to pH 8.5 with 100 mM HCl.)
Flow rate: 1
Injection volume: 20
Detector: F ex 340 em 460 following post-column reaction. The column effluent flowed through a 3 m × 0.51 mm ID stainless steel tube at 140° to the detector. (Although the reagents are in the mobile phase the derivatization reaction does not take place until the post-column reactor where the insecticides are hydrolyzed to methylamine that is then derivatized. This procedure avoids the use of a second pump for the post-column reagent.)

CHROMATOGRAM
Retention time: 14
Limit of detection: 400 pg

OTHER SUBSTANCES
Simultaneous: aldicarb, butocarboxim, carbaryl, carbofuran, dioxacarb, methomyl

KEY WORDS
post-column reaction

REFERENCE
Sabala,A.; Portillo,J.L.; Broto-Puig,F.; Comellas,L. Development of a new high-performance liquid chromatography method to analyse N-methylcarbamate insecticides by a simple post-column derivatization system and fluorescence detection, *J.Chromatogr.A*, **1997**, *778*, 103–110.

SAMPLE
Matrix: tissue
Sample preparation: 21 g Liver + 60 g anhydrous sodium sulfate, mix with spatula, add 200 mL dichloromethane, mix with spatula, homogenize (VirTis 45) for 2 min at medium speed, filter through 5 g anhydrous sodium sulfate, re-extract tissue and sodium sulfate with 100 mL dichloromethane, filter, wash out flask with 25 mL dichloromethane, filter. Combine filtrates and filter them through 2 g anhydrous sodium sulfate, rinse flask with 20 mL dichloromethane, wash filter with 10 mL dichloromethane. Concentrate filtrate to 1-2 mL under reduced pressure at 30° (do not allow to go dry), transfer residue to a tube with 1-2 mL cyclohexane, wash in with dichloromethane:cyclohexane 50:50, make volume in tube 7.5 mL, filter (0.45 μm), add 5 mL to a 600 × 25 tube containing 60 g 200-400 mesh BioBeads SX-3 resin (Analytical Bio-Chemistry Laboratories), pump through at 5 mL/min with dichloromethane:cyclohexane 50:50 mobile phase, collect fraction containing the compound, evaporate under reduced pressure at 30° to about 1 mL, make up to 2 mL with dichloromethane, add 1 mL to a 1 mL 100 mg Bond Elut aminopropyl SPE cartridge (previously conditioned with 1 mL dichloromethane), elute with 3-5 mL dichloromethane:MeOH 98.5:1.5, evaporate eluate to dryness at 30° under reduced

pressure (do not over dry), reconstitute in 200 μL MeOH, vortex for 5 s, filter (0.45 μm), inject a 20-30 μL aliquot.

HPLC VARIABLES
Guard column: Guard-PAK (Waters no. 88070)
Column: 250 × 4.6 5 μm Zorbax C8
Mobile phase: Gradient. MeCN:water from 12:88 to 70:30 over 30 min, to 80:20 over 1 min, maintain at 80:20 for 8 min, re-equilibrate at initial conditions for 10 min.
Flow rate: 1.5
Injection volume: 20-30
Detector: F ex 340 em 418, following post-column derivatization. The column effluent is mixed with 50 mM NaOH at 0.27 mL/min and the mixture flows through a 1 mL coil at 80° and is mixed with 140 μg/mL o-phthalaldehyde and 1 mL/L mercaptoethanol in 50 mM pH 10.5 potassium borate buffer pumped at 0.27 mL/min. The mixture flows through a 1 mL coil at 40° to the detector.

CHROMATOGRAM
Retention time: 22.1
Limit of quantitation: 5 ppb

OTHER SUBSTANCES
Extracted: aldicarb, bendiocarb, bufencarb, carbaryl, carbofuran, dioxacarb, isoprocarb, methiocarb, methomyl, oxamyl, promecarb

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KEY WORDS
liver; pig; cow; duck; SPE; post-column reaction

REFERENCE
Ali,M.S.; White,J.D.; Bakowski,R.S.; Stapleton,N.K.; Williams,K.A.; Johnson,R.C.; Phillippo,T.; Woods,R.W.; Ellis,R.L. Extension of a liquid chromatographic method for *N*-methylcarbamate pesticides in cattle, swine, and poultry liver, *J.AOAC Int.*, **1993**, *76*, 907–910.

SAMPLE
Matrix: water
Sample preparation: Extract 500 mL water with two 25 mL portions of dichloromethane, combine the extracts and dry them over anhydrous sodium sulfate for 10 min, evaporate to dryness under a stream of air, reconstitute with 40 μL acetone, add 300 μL 100 mM sodium carbonate, heat at 45-50° for 30-40 min, cool, add 300 μL acetone, add 100 μL 0.2% dansyl chloride in acetone, mix well, heat at 45° for 20 min, cool, evaporate the acetone under a stream of air, extract with 300 μL benzene (Caution! Benzene is a carcinogen!). Remove the organic layer and dry it over anhydrous sodium sulfate, inject a 1-10 μL aliquot.

HPLC VARIABLES
Column: 1000 × 2.4 Zipax coated with 0.5% β,β'-oxydipropionitrile
Mobile phase: Hexane:EtOH 95:5
Injection volume: 1-10
Detector: F primary filter Turner 810, secondary filter Turner 827

CHROMATOGRAM
Retention time: k' 0.67

OTHER SUBSTANCES
Simultaneous: aldicarb (Temik), carbaryl (Sevin), carbofuran, Carzol, dimethylamine, methomyl, methylamine, Mobam

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KEY WORDS
lake water; derivatization

REFERENCE
Frei,R.W.; Lawrence,J.F.; Hope,J.; Cassidy,R.M. Analysis of carbamate insecticides by fluorigenic labelling and high-speed liquid chromatography, *J.Chromatogr.Sci.*, **1974**, *12*, 40–44.

SAMPLE
Matrix: water
Sample preparation: Filter, inject a 400 μL aliquot of the filtrate.

HPLC VARIABLES
Guard column: C18
Column: 150 × 4.6 3 μm HS-3C18 (Perkin Elmer)
Mobile phase: Gradient. MeCN:water from 5:95 to 20:80 over 13 min, to 65:35 over 15 min, return to initial conditions over 2 min, re-equilibrate at initial conditions for 8 min.
Flow rate: 1
Injection volume: 400
Detector: F ex 340 em 460 following post-column reaction. The column effluent mixed with the reagent pumped at 0.1 mL/min and the mixture flowed through a 500 μL reaction coil at 95° to the detector. (Prepare the reagent by adding 1.25 mL 10 M NaOH to 100 mL water, add 10 mL 18 mg/mL N,N-dimethyl-2-mercaptoethylamine hydrochloride (Thiofluor; Pickering Laboratories, Mountain Vie CA), add 2.5 mL 10 mg/mL o-phthalaldehyde in MeOH, make up to 250 mL with water, filter (0.45 μm nylon), degas with helium for 10 min before use. Prepare fresh each day.)

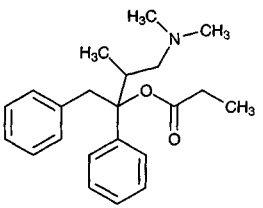
CHROMATOGRAM
Retention time: 28.35
Internal standard: 4-bromo-3,5-dimethylphenyl N-methylcarbamate (34)
Limit of detection: 0.6 ng/mL

OTHER SUBSTANCES
Simultaneous: aldicarb, aldicarb sulfone, aldicarb sulfoxide, carbaryl, carbofuran, 3-hydroxycarbofuran, methiocarb, methomyl, oxamyl

KEY WORDS
post-column reaction

REFERENCE
Simon,V.A.; Pearson,K.S.; Taylor,A. Determination of N-methylcarbamates and N-methylcarbamoyloximes in water by high performance liquid chromatography with the use of fluorescence detection and a single o-phthalaldehyde post-column reaction, *J.Chromatogr.*, **1993**, *643*, 317–320.

Propoxyphene



Molecular formula: $C_{22}H_{29}NO_2$
Molecular weight: 339.48
CAS Registry No.: 469-62-5, 1639-60-7 (HCl), 26570-10-5 (napsylate monohydrate), 55557-30-7 (l-form napsylate monohydrate), 2338-37-6 (l form), 17140-78-2 (l-form napsylate anhydrous)
Merck Index: 8024
Lednicer No.: 1 50, 298; 2 57

SAMPLE
Matrix: blood
Sample preparation: 2 mL Whole blood or plasma + 2 mL buffer + 5 mL chloroform:isopropanol: n-heptane 60:14:26, shake gently horizontally for 10 min, centrifuge at 2800 g for 10 min. Remove the lower organic layer and evaporate it to dryness under vacuum at 45°, reconstitute the residue in 100 μL mobile phase, centrifuge at 2800 g for 5 min, inject a 50 μL aliquot of the supernatant. (Buffer was saturated ammonium chloride solution 25% diluted with water, adjusted to pH 9.5 with 25% ammonia solution.)

HPLC VARIABLES
Column: 300 × 3.9 4 μm NovaPack C18

Mobile phase: MeOH:THF:buffer 65:5:30 (Buffer was 0.68 g/L (10 mM (sic)) KH_2PO_4 adjusted to pH 2.6 with concentrated orthophosphoric acid.) (At the end of each session wash the column with water for 1 h and MeOH for 1 h, re-equilibrate for 30 min.)
Column temperature: 30
Flow rate: 0.8
Injection volume: 50
Detector: UV 259

CHROMATOGRAM
Retention time: 7.24
Limit of detection: <120 ng/mL

KEY WORDS
whole blood; plasma; interferences may occur–compounds (all of which are extracted) elute in this order tenoxicam; iproniazid; methocarbamol; methotrexate; caffeine; nialamide; colchicine; cytarabine; benzoylecgonine; acetaminophen; diazoxide; dacarbazine; sulfinpyrazole; flumazenil; sulpride; morphine; atenolol; toloxatone; terbutaline; albuterol; phenobarbital; ranitidine; tiapride; phenol; chlormezanone; aspirin; metformin; ritodrine; codeine; sultopride; amisulpride; naltrexone; lisinopril; benzocaine; nizatidine; nalorphine; mephenesin; naloxone; sotalol; carteolol; procainamide; carbamazepine; bromazepam; nalbuphine; nadolol; procarbazine; dihydralazine; omeprazole; strychnine; acebutolol; glutethimide; chlorpropamide; glipizide; triazolam; prazosin; flunitrazepam; clonazepam; metoclopramide; melphalan; estazolam; tolbutamide; ephedrine; clonidine; pindolol; clobazam; minoxidil; disopyramide; nitrazepam; dextromethorphan; tofisopam; zopiclone; debrisoquine; sulindac; alprazolam; cycloguanil; lorazepam; methaqualone; ketamine; piroxicam; metoprolol; nifedipine; quinine; mephentermine; prilocaine; pentazocine; oxazepam; tiaprofenic acid; quinidine; celiprolol; ajmaline; yohimbine; lidocaine; secobarbital; viloxazine; mepivacaine; meperidine; doxylamine; labetalol; temazepam; amodiaquine; benperidol; droperidol; hydroxychloroquine; zolpidem; ketoprofen; alminoprofen; cicletanine; moclobemide; chloroquine; cocaine; timolol; nomifensine; ticlopidine; acenocoumarol; vindesine; mexiletine; dipyridamole; trazodone; pipamperone; pyrimethamine; benazepril; vincristine; metapramine; chlordiazepoxide; oxprenolol; warfarin; clorazepate; flecainide; phencyclidine; thiopental; fenfluramine; metipranolol; triprolidine; naproxen; buprenorphine; verapamil; buspirone; tianeptine; midazolam; bupivacaine; carbinoxamine; loprazolam; cetirizine; chlorpheniramine; moperone; cibenzoline; medifoxamine; astemizole; vinblastine; nicardipine; bisoprolol; diltiazem; glibornuride; reserpine; aconitine; nitrendipine; diazepam; mianserin; ramipril; haloperidol; tetracaine; alprenolol; acepromet azine; glibenclamide; chlorophenacinone; doxepin; nimodipine; diphenhydramine; cyclizine; histapyrrodine; phenylbutazone; demexiptiline; clozapine; proguanil; trifluperidol; medazepam; cyamemazine; bumadizone; suriclone; propranolol; acepromazine; dothiepin; dextromoramide; fenoprofen; dextropropoxyphene; loxapine; betaxolol; propafenone; promethazine; thioproperazine; methadone; amoxapine; quinupramine; opipramol; cyproheptadine; brompheniramine; mefenidramine; protriptyline; flurbiprofen; tetrazepam; zorubicin; prazepam; alimemazine; loperamide; imipramine; desipramine; levomepromazine; hydroxyzine; niflumic acid; penbutolol; fluvoxamine; pimozide; daunorubicin; indomethacin; maprotiline; tropatenine; etodolac; fluoxetine; amitriptyline; nortriptyline; tioclomarol; diclofenac; mefloquine; trimipramine; chlorambucil; lidoflazine; ibuprofen; floctafenine; alpidem; loratadine; chlorpromazine; clomipramine; carpipramine; thioridazine; fentiazac; clemastine; mefenamic acid; fluphenazine; prochlorperazine; penfluridol; bepridil; terfenadine; trifluoperazine

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REFERENCE
Tracqui,A.; Kintz,P.; Mangin,P. Systematic toxicological analysis using HPLC/DAD, *J.Forensic Sci.*, **1995**, *40*, 254–262.

SAMPLE
Matrix: blood, urine
Sample preparation: Add 1 mL whole blood or urine to Toxi-Tube A (Toxi-Lab, Irvine CA), add 3 mL water, mix by gentle inversion for 5 min, centrifuge at 1500 g for 5 min. Remove the organic layer and evaporate it to dryness under a stream of nitrogen at 40°, reconstitute the residue with 50 µL MeCN:water 50:50, vortex for 10 s, centrifuge at 7500 g for 2 min, inject a 10 (urine) or 30 (blood) µL aliquot. (The detector wavelength shown is the wavelength of maximum absorbance. This will not necessarily be the optimal wavelength for the separation. Multiple wavelengths from 200-350 nm can be scanned using a diode-array detector. Otherwise, 220 nm may be a reasonable choice for initial work. Matrix may interfere.)

HPLC VARIABLES

Guard column: 20 mm long Symmetry C18
Column: 250 × 4.6 5 μm Symmetry C8 (Waters)
Mobile phase: Gradient. A was 50 mM pH 3.8 sodium phosphate buffer. B was MeCN. A:B 85:15 for 6.5 min, 65:35 for 18.5 min, 20:80 for 3 min (step gradient), re-equilibrate at initial conditions for 7 min.
Column temperature: 30
Flow rate: 1 for 6.5 min, to 1.5 over 18.5 min, maintain at 1.5 for 3 min (re-equilibrate at 1.5 mL/min)
Injection volume: 10-30
Detector: UV 200.5

CHROMATOGRAM

Retention time: 15.82

KEY WORDS

whole blood

REFERENCE

Gaillard,Y.; Pépin,G. Use of high-performance liquid chromatography with photodiode-array UV detection for the creation of a 600-compound library. Application to forensic toxicology, *J.Chromatogr.A*, **1997**, *763*, 149–163.

SAMPLE

Matrix: solutions
Sample preparation: Prepare a 10 μg/mL solution in MeOH, inject a 20 μL aliquot.

HPLC VARIABLES

Column: 125 × 4.9 Spherisorb S5W silica
Mobile phase: MeOH containing 10 mM ammonium perchlorate and 1 mL/L 100 mM NaOH in MeOH, pH 6.7
Flow rate: 2
Injection volume: 20
Detector: E, LeCarbone, V25 glassy carbon electrode, + 1.2 V

CHROMATOGRAM

Retention time: 2.5

OTHER SUBSTANCES

Also analyzed: acebutolol, acepromazine, acetophenazine, N-acetylprocainamide, albuterol, alprenolol, amethocaine, amiodarone, amitriptyline, antazoline, atenolol, azacyclonal, bamethan, benactyzine, benperidol, benzethidine, benzocaine, benzoctamine, benzphetamine, benzquinamide, bromhexine, bromodiphenhydramine, bromperidol, brompheniramine, brompromazine, buclizine, bufotenine, bupivacaine, buprenorphine, butacaine, butethamate, chlorcyclizine, chlorpheniramine, chlorphenoxamine, chlorprenaline, chlorpromazine, chlorprothixene, cimetidine, cinchonidine, cinnarizine, clemastine, clomipramine, clonidine, cocaine, cyclazocine, cyclizine, cyclopentamine, cyproheptadine, deserpidine, desipramine, dextromoramide, dicyclomine, diethylcarbamazine, diethylpropion, diethylthiambutene, dihydroergotamine, dimethindene, dimethothiazine, diphenhydramine, diphenoxylate, dipipanone, diprenorphine, dipyridamole, disopyramide, dothiepin, doxapram, doxepin, doxylamine, droperidol, ephedrine, ergocornine, ergocristine, ergocristinine, ergocryptine, ergometrine, ergosine, ergosinine, ergotamine, ethopropazine, etorphine, etoxeridine, fenethazine, fenfluramine, fenoterol, fentanyl, flavoxate, fluopromazine, flupenthixol, fluphenazine, flurazepam, haloperidol, hydroxyzine, hyoscine, ibogaine, imipramine, indapamine, iprindole, isothipendyl, isoxsuprine, ketanserin, laudanosine, lidocaine, lofepramine, loxapine, maprotiline, mecamylamine, meclophenoxate, meclozine, medazepam, mephentermine, mepivacaine, meptazinol, mepyramine, mesoridazine, metaraminol, methadone, methamphetamine, methapyrilene, methdilazene, methotrimeprazine, methoxamine, methoxyphenamine, methoxypromazine, methylephedrine, methylergonovine, methysergide, metoclopramide, metopimazine, metoprolol, mianserin, morazone, nadolol, nalorphine, naloxone, naphazoline, nicotine, nifedipine, nomifensine, nortriptyline, noscapine, orphenadrine, oxeladin, oxprenolol, oxymetazolin, papaverine, pargyline, pecazine, penbutolol, pentazocine, penthienate, pericyazine, perphenazine, phenadoxone, phenampromide, phena-

zocine, phenbutrazate, phendimetrazine, phenelzine, phenglutarimide, phenindamine, pheniramine, phenmetrazine, phenomorphan, phenoperidine, phenothiazine, phenoxybenzamine, phentolamine, phenylephrine, phenyltoloxamine, physostigmine, piminodine, pimozide, pindolol, pipamazine, pipazethate, piperacetazine, piperidolate, pipradol, pirenzepine, piritramide, pizotifen, practolol, pramoxine, prazosin, prenylamine, prilocaine, primaquine, proadifen, procainamide, procaine, prochlorperazine, procyclidine, proheptazine, prolintane, promazine, promethazine, pronethalol, properidine, propiomazine, propranolol, prothipendyl, protriptyline, proxymetacaine, pseudoephedrine, pyrimethamine, quinidine, quinine, ranitidine, rescinnamine, sotalol, tacrine, terazosin, terbutaline, terfenadine, thenyldiamine, theophylline, thiethylperazine, thiopropazate, thioproperazine, thioridazine, thiothixene, thonzylamine, timolol, tocainide, tolpropamine, tolycaine, tranylcypromine, trazodone, trifluoperazine, trifluperidol, trimeperidine, trimeprazine, trimethobenzamide, trimethoprim, trimipramine, tripelennamine, triprolidine, tryptamine, verapamil, xylometazoline

REFERENCE

Jane,I.; McKinnon,A.; Flanagan,R.J. High-performance liquid chromatographic analysis of basic drugs on silica columns using non-aqueous ionic eluents. II. Application of UV, fluorescence and electrochemical oxidation detection, *J.Chromatogr.*, **1985**, *323*, 191–225.

SAMPLE

Matrix: solutions

HPLC VARIABLES

Column: 250 × 4.6 Zorbax RX

Mobile phase: Gradient. A was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 1 L water. B was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 200 mL water, make up to 1 L with MeCN. A:B from 100:0 to 0:100 over 30 min, maintain at 0:100 for 5 min.

Column temperature: 30

Flow rate: 2

Detector: UV 210

OTHER SUBSTANCES

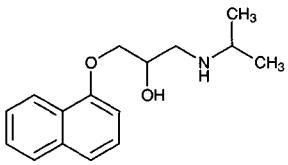
Also analyzed: acepromazine, acetaminophen, acetophenazine, albuterol, aminophylline, amitriptyline, amobarbital, amoxapine, amphetamine, amylocaine, antipyrine, aprobarbital, aspirin, atenolol, atropine, avermectin, barbital, benzocaine, benzoic acid, benzotropine, benzphetamine, berberine, bibucaine, bromazepan, brompheniramine, buprenorphine, buspirone, butabarbital, butacaine, butethal, caffeine, carbamazepine, carbromal, chloramphenicol, chlordiazepoxide, chloroquine, chlorothiazide, chloroxylenol, chlorphenesin, chlorpheniramine, chlorpromazine, chlorpropamide, chlortetracycline, cimetidine, cinchonidine, cinchonine, clenbuterol, clonazepam, clonixin, clorazepate, cocaine, codeine, colchicine, cortisone, coumarin, cyclazocine, cyclobenzaprine, cyclothiazide, cyheptamide, cymarin, danazol, danthron, dapsone, debrisoquine, desipramine, dexamethasone, diamorphine, diazepam, diclofenac, diethylpropion, diethylstilbestrol, diflunisal, digitoxin, digoxin, diltiazem, diphenhydramine, diphenoxylate, diprenorphine, dipyrone, disulfiram, dopamine, doxapram, doxepin, dronabinol, ephedrine, epinephrine, epinine, estradiol, estriol, estrone, ethacrynic acid, ethosuximide, etonitazene, etorphine, eugenol, famotidine, fenbendazole, fencamfamine, fenoprofen, fenproporex, fentanyl, flubendazole, flufenamic acid, flunitrazepam, 5-fluorouracil, fluoxymesterone, fluphenazine, furosemide, gentisic acid, gitoxigenin, glipizide, glunixin, glutethimide, glybenclamide, guaiacol, halazepam, haloperidol, hydrochlorothiazide, hydrocodone, hydrocortisone, hydromorphone, hydroxyquinoline, ibogaine, ibuprofen, iminostilbene, imipramine, indomethacin, isocarbostyril, isocarboxazid, isoniazid, isoproterenol, isoxsuprine, ivermectin, ketamine, ketoprofen, kynurenic acid, levorphanol, lidocaine, lorazepam, lormetazepam, loxapine, mazindol, mebendazole, meclizine, meclofenamic acid, medazepam, mefenamic acid, megestrol, mepacrine, meperidine, mephentermine, mephenytoin, mephesin, mephobarbital, mepivacaine, mescaline, mesoridazine, methadone, methamphetamine, methapyrilene, methaqualone, methazolamide, methocarbamol, methoxamine, methsuximide, methyl salicylate, methyldopa, methyldopamine, methylphenidate, methylprednisolone, methyltestosterone, methyprylon, metoprolol, mibolerone, morphine, nadolol, nalorphine, naloxone, naltrexone, naphazoline, naproxen, nefopam, niacinamide, nicotine, niacin, nifedipine, niflumic acid, nitrazepam, norepinephrine, nortriptyline, noscapine, nylidrin, oxazepam, oxycodone, oxymorphone, oxyphenbutazone, oxytetracycline, papaverine, pargyline, pemoline, pentazocine, pentobarbital, persantine, phenacetin, phenazocine, phenazopyridine, phencyclidine, phendimetrazine, phenelzine, pheniramine, phenobarbital, phenothiazine, phensuximide, phentermine, phenyl-

butazone, phenylephrine, phenylpropanolamine, piperocaine, prazepam, prednisolone, primidone, probenecid, progesterone, propiomazine, propranolol, propylparaben, pseudoephedrine, puromycin, pyrilamine, pyrithyldione, quazepam, quinaldic acid, quinidine, quinine, ranitidine, recinnamine, reserpine, resorcinol, saccharin, albuterol, salicylamide, salicylic acid, scopolamine, scopoletin, secobarbital, strychnine, sulfacetamide, sufadiazine, sulfadimethoxine, sulfaethidole, sulfamerazine, sulfamethazine, sulfamethoxizole, sulfanilamide, sulfapyridine, sulfasoxizole, sulindac, tamoxifen, temazepam, testosterone, tetracaine, tetracycline, tetramisole, thebaine, theobromine, theophylline, thiabendazole, thiamine, thiamylal, thiobarbituric acid, thioridazine, thiosalicylic acid, thiothixene, thymol, tolazamide, tolazoline, tobutamide, tolmetin, tranylcypromine, triamcinolone, tribenzylamine, trichloromethiazide, trifluoperazine, trihexyphenidyl, trimethoprim, tripelennamine, triprolidine, tropacocaine, tyramine, verapamil, vincamine, warfarin, yohimbine, zoxazolamine

REFERENCE

Hill,D.W.; Kind,A.J. Reversed-phase solvent gradient HPLC retention indexes of drugs, *J.Anal.Toxicol.*, **1994**, *18*, 233–242.

Propranolol



Molecular formula: $C_{16}H_{21}NO_2$
Molecular weight: 259.35
CAS Registry No.: 525-66-6, 318-98-9 (HCl)
Merck Index: 8025
Lednicer No.: 1 117; 2 105, 212

SAMPLE

Matrix: bile, perfusate
Sample preparation: 500 μL Perfusate or 100 μL bile + 50 μL 50 μg/mL labetalol + 1 mL 1 M pH 10.3 carbonate buffer + 5 mL acid-washed diethyl ether, vortex, centrifuge. Remove the organic layer and add it to 125 μL 0.5% phosphoric acid, extract, inject a 10 μL aliquot of the aqueous layer. (Deconjugate 500 μL perfusate with 250 μL 8000 U/mL β-D-glucuronidase/aryl sulfatase in 200 mM pH 4.5 sodium acetate buffer, heat at 40° for 1 h, proceed as above.)

HPLC VARIABLES

Column: 100 × 8 4 μm Novapak phenyl radial compression
Mobile phase: MeCN:water:triethylamine 23:77:1 adjusted to pH 3.6 with concentrated phosphoric acid
Flow rate: 3
Injection volume: 10
Detector: F ex 295 em 360

CHROMATOGRAM

Retention time: 5.5
Internal standard: labetalol (6.9)
Limit of quantitation: 62.5 ng/mL

KEY WORDS

sheep; liver; pharmacokinetics

REFERENCE

Ring,J.A.; Ghabrial,H.; Ching,M.S.; Shulkes,A.; Smallwood,R.A.; Morgan,D.J. Fetal hepatic propranolol metabolism. Studies in the isolated perfused fetal sheep liver, *Drug Metab.Dispos.*, **1995**, *23*, 190–196.

SAMPLE

Matrix: blood
Sample preparation: Prepare a silica SPE cartridge. Fill 3 mL cartridge with 500 mg Silica gel 60 (Merck). Condition it with 2.5 mL MeOH and with 2.5 mL water. Add 500 μL plasma or serum to the SPE cartridge. Wash with 1 mL water, elute with 3 mL MeOH (added dropwise).